Scanning probe methods for studying surface structures - history of development and recent possibilities

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The idea of using solid-state probes for recording and reproducing information was born at the end of the 19th century by the invention of a gramophone. In 1929 the German engineer Schmalz G. made the first probe microscope Stylus Profiler with which it was possible to obtain a surface image of 11x18 mm with an increase of 1000 times. The image was recorded on a photographic plate. These developments were developed in the direction of creating co-ordinate profilometers with profile registration by piezoelectric cantilevers. Profilometers of similar type are used in industry today.

High-resolution probe technology for visualization of surface structures began to be developed since 1966. At the Institute of Standards in the US Russell Young group made a device Topographer which was able to see atomic steps. Personal computers were still in their infancy and had a small memory, so the mapping of topography was carried out using a two-coordinate recorder. The first exceptionally important, key innovation proposed by the Russell Young group was the use of piezoelectric ceramics for movement of the tip and sample.

The historical period in the development of scanning probe microscopy began in 1971 in the group of Heinrich Rohrer and Helmut Binnig in the IBM Tsurikh branch from the target installation for obtaining atomic resolution (the Nobel Prize in 1986).

G. Binnig and G. Rohrer showed that using an instrument called a scanning tunnel microscope, whose schematic diagram is similar to the Young topographer, allows one to obtain images of individual atoms. Personal computers were used to control the device and to process the results. Progress in the capabilities of devices is determined by the power of the used computers. In 1986, the same group proposed the use of a tubular piezoceramic scanner. To record the surface relief, it was suggested to use flexible beams with a sharp needle on the loose end - cantilevers, and instruments for recording the relief were called atomic-force microscopes (AFM). In the AFM of Binnig, Kwait and Gerber, a tunnel sensor was used to record the normal displacement of the cantilever, which is extremely inconvenient. A powerful innovation that made AFM a reality was Ammer and Maier's invention of an opto-positional scheme for recording the change in the angles of cantilever inclination, when a four-section photodiode is used as a recording element, it is possible to record both normal and lateral interaction forces probe and the test surface of the sample.

Innovation to prevent the effect of the capillary was born in 1986. It was proposed by Binnig, his real possibilities are shown by Ducker, Cook and Clarke is integrated into the first industrial atomic force microscopes by Vergil Eling called "tipping" or semi-contact mode.

In 1987, a group of researchers were asked to make cantilevers using the silicon technology, where the selective etching both "grooves" and tips with an angle at the apex determined by the properties of the crystal were already developed. The beams thickness could be set either by the coating thickness or by the depth of alloying with boron or phosphorus. The ability to manufacture cantilevers using the methods of microelectronic technologies made them an affordable consumable and ensured the possibility of wide distribution of the method.

In the late eighties and early nineties, the possibility of recording a number of surface physical properties in various conditions, from ultrahigh vacuum to liquid, and methods for modifying the surface (SPM lithography) was realized, which are now integrated into all scanning probe microscopes (SPM) under several different names.

In the late 1980s, the work on the creation of SPM was also launched in the USSR. A corporation MDT started to create SPMs with the capabilities of lithographers, where it would be possible to carry out both research and modification of the properties of molecular structures. By 1995, AFMs were created, and by 1997 - multi-mode instruments for complex studies of surface structures without destroying in the process of investigation.

To date, scanning probe microscopy has become part of the classical methods of studying nanostructures and is widely used for qualitative assessment of physical and chemical properties and geometrical parameters of the surfaces.

In addition to topography, SPMs can measure a distribution of many physical properties: surface electric potential; surface conductivity; electrical capacity of the probe-surface system; magnetic forces in a system of probe and given magnetic surface; piezoelectric properties; thermal conductivity; mechanical properties (Young's modulus, hardness); adhesion properties. Measurements can be made in air, in a gaseous atmosphere, in a liquid and in a vacuum in wide temperature range from superlow to several hundred degrees Celsius. The modern possibilities of computer technology and digital technologies allow to reduce significantly the requirements for the user's qualification.

The training of specialists is an exceptionally important task for development of modern technology which must begin at a school bench. The ability to see and actively influence molecular structures changes dramatically and intensifies the depth of understanding of physics, chemistry, and biology. Fig. 1 shows the last version of the NANOEDUCATOR device, which currently equips dozens of educational classes in Russia and abroad. This device was among the best world products according to the version of Research & Developments magazine in 2011.

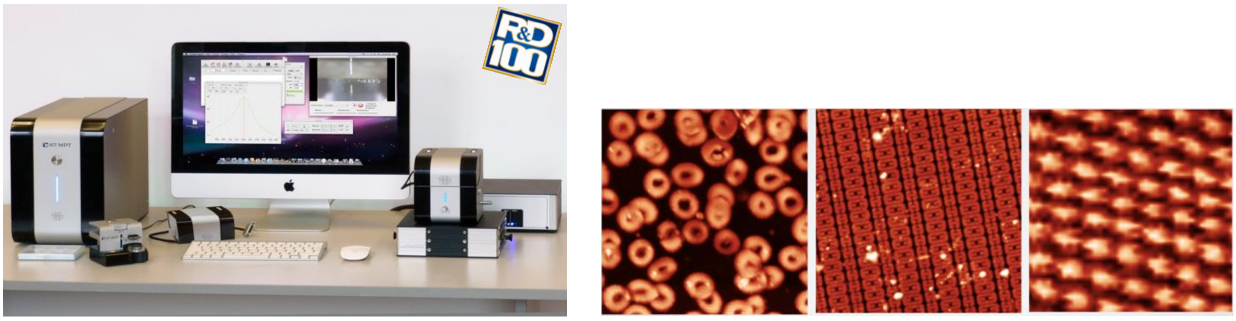

Figure 1. The education device NANOEDUCATOR-II and the obtained images of erythrocytes (image area 50x50 μm), section of microcircuit (30x30 μm), atomic structure of highly oriented pyrolitic graphite (2x2 nm).

It should be noted that SPMs are devices that are very sensitive to acoustic disturbances, changes of temperature and humidity. To minimize these factors, it is necessary to operate inside acoustically protected boxes equipped with an active or passive vibro-protection system, a system for maintaining temperature and humidity of high accuracy. The provision of all these conditions has been solved for the entire range of NT-MDT SPMs, providing a drift of less than 10 nm/h, which allows to obtain atomic resolution at relatively low scanning frequencies.

A powerful development was obtained by combines of SPMs and spectrometers, combining methods of high-resolution measurements of topography and various physical properties of surface structures. Instruments allow to obtain information both on the physical properties of surface structures and on the qualitative composition from luminescence spectroscopy, Raman spectroscopy and high-resolution IR spectroscopy.

The consistent innovative development of SPMs allowed re-positioning these devices, significantly reducing the requirements for users from the entourage of the method, to specialists in probe microscopy, and at present the devices of the latest developments of the NT-MDT Spectrum Instruments group can be successfully used by laboratory technicians and engineers to monitor the technological parameters of processes, and materialists, whose goal is to obtain well-interpreted information on physical and physicochemical properties of different structures.